Influence of Al on Shape Memory Effect and Twinning Induced Plasticity of Fe-Mn-Si-Al System Alloy*

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We have investigated the shape memory effect and ductility of Fe-30Mn-(6-x)Si-xAl (x = 0, 1, 2 and 3; in mass-%) alloys. The alloy with x = 0 shows a good shape memory effect but suffers from poor ductility, while the alloys with x = 2 and 3 are well-known TWIP (twinning induced plasticity) steels that show high ductility. In the present study, it was found that the shape memory effect is observed in the samples with x = 0 and 1, but disappears when x exceeds 2. On the other hand, the ductility almost linearly increases with increasing the amount of Al. A good combination of the shape memory effect and ductility was achieved in the alloy with x = 1.

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1. Introduction

Shape memory alloys (SMAs) such as Ni-Ti intermetallic compounds, Cu and Fe based alloys are applied for various engineering applications like actuators.¹ The applications of ferrous shape memory alloys with good workability and weldability have been widely investigated.²,³ In particular, Fe-Mn-Si based alloys⁴,⁵ have drawn attention as structural materials because of a high cost performance and a combination of good shape memory effect and mechanical properties.⁶

The shape memory effect (SME) of Fe-Mn-Si based alloys is ascribed to the reversed transformation of stress-induced ε (hcp) martensite to the γ (fcc) austenite on heating, which is not reversible and has a large thermal hysteresis. The alloys therefore have been used for pipe joint in structural materials,⁷,⁸ rail coupling,⁹ pre-stressed concrete,¹⁰,¹¹ where reversible motion is not requisite. Typical chemical compositions of Fe-Mn-Si ternary alloys are in the range of 14~33 mass%Mn, and 5~6 mass%Si. To impart corrosion resistance, 5% of Cr is commonly added to the alloy.⁵ A recoverable strain of a conventional Fe-Mn-Si alloy is about 2%. This value can be increased to about 4% either by training treatment in that deformation and heating is repeated,¹² or by dispersing fine precipitates of NbC or VN.¹³~¹⁶ One drawback of the alloy was its poor ductility, which is only 30% at room temperature, because of high work hardening.

Recent studies have shown that high manganese steels exhibit a combination of high strength, high ductility and high toughness by utilizing transformation-induced plasticity or twinning-induced plasticity. They are called TRIP or TWIP steels and are actively studied in the field of structural materials for automobile engineering.¹⁷,¹⁸ In particular, TWIP steel with the composition of Fe-30Mn-3Si-3Al (hereinafter compositions are shown in mass%) shows a large ductility of about 90%, for which the product of tensile strength and ductility (RmA value) reached 50000 MPa%.¹⁷ The composition of this alloy is such that a part of Si in Fe-30Mn-6Si shape memory alloy is replaced by Al, although there is no report on the shape memory effect.

In this study, we noticed a similarity in the chemical compositions between SMA and TWIP steel. We then prepared four kinds of Fe-Mn-Si-Al alloys with different Si and Al contents with the aim of investigating the effect of Al on the SME and TWIP effect.

2. Experimental Procedures

2.1 Sample preparation

The alloys with chemical compositions of Fe-30Mn-(6-x)Si-xAl (x = 0, 1, 2, 3) were prepared by vacuum induction melting. In this paper, hereafter the alloys were referred to as Al-0, Al-1, Al-2, Al-3 using mass% of Al. It is known that the sample Al-0 exhibits SME,⁵ and the samples Al-2 and Al-3 have TWIP effect.¹⁷ The specimens were solution-treated at 1150°C for 10 h followed by air quenching or at 1000°C for 3 h followed by water quenching after hot rolling and forging at 1000°C. We measured shape memory effect and ductility using specimens cut with electric spark machining.

2.2 Shape memory effect and mechanical properties

Shape memory effect was characterized with bending tests. Plate-like specimens with dimensions of 1.6 × 0.8 × 18.0 mm³ were bent around a ring jig of 12.0 mm diameter, and were heated at 600°C for 10 min. The magnitude of shape recovery was measured by comparing the change in shapes taken with a digital camera. The shape recovery ratio was determined using the following equation by measuring curvature radius before and after heating (see Fig. 1).

\[ \varepsilon = \frac{t}{2r}, \quad R = \left( \frac{\varepsilon_2 - \varepsilon_1}{\varepsilon_1} \right) \]

\( \varepsilon \): the maximum strain of sample surface (suffix 1 and 2 correspond to before and after heating, respectively.),
\( t \): thickness, \( r \): curvature radius, \( R \): shape recovery ratio.

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The ductility and strength were measured with tensile tests at a strain rate of 0.1 mm/min for specimens with dimensions of 4.0 × 3.0 × 20.0 mm³. The ductility was determined by measuring the gauge length before and after the tensile test. For some specimens, stress-strain (SS) curves of 0̶10% were measured using a strain gauge.

2.3 Microstructure

Microstructural observations were performed with an optical microscope, a transmission electron microscope (TEM) and an atomic force microscope (AFM).

For optical microscopy, the samples of Al-0̶Al-3 were mechanically polished to obtain smooth surfaces. The surface relieves formed by 3% tensile deformation were observed using a differential interference microscope.

In addition, TEM observations of Al-1 specimens deformed by about 2% tensile strain were performed to analyze the deformation microstructure, since the sample showed good shape memory effect and high ductility. The specimens for TEM observation were carefully prepared to avoid the formation of stress-induced martensite and reversed transformation on heating. The thickness of the specimen was reduced from 0.85 mm to 0.2 mm using a chemical polishing solution of hydrogen peroxide and hydrofluoric acid mixed in the ratio of 10 : 1. The disk samples of 3 mm diameter were then formed using the same chemical polishing solution. The specimens were further subjected to electropolishing using acetic acid and perchloric acid mixed in the ratio of 20 : 1 at room temperature.

AFM measurements were also performed to analyze quantitatively the surface relieves of the sample Al-1. The samples for AFM observations were electropolished to obtain clean surfaces after mechanical polishing. The observations were made for the samples at the tensile strain of ε = 3%, 10% and after heating.

3. Results and Discussion

3.1 Shape memory effect and mechanical properties

The results of SME tests are shown in Fig. 2. Left figures show the samples after bending and right figures are for those after heating. One can see that the shape recovery of the samples Al-2 and Al-3 was almost negligible. In contrast, the sample Al-1 showed a similar SME to Al-0.

Table 1 shows the values of shape recovery ratios that were calculated based on the change in the curvature radius. The samples Al-0 and Al-1 showed the shape recovery ratios exceeding 40%, while the samples Al-2 and Al-3 showed small recovery values of 3% and 0%.

The ductility, the tensile strength and RmA-values are also shown in Table 1. It is clear that the ductility increases with increasing the amount of Al. Thus, the ductility of Al-1 was lower than those of Al-2, Al-3 or TWIP steels, but it was greater than that of Al-0 or conventional SMA. The tensile strength decreased with increasing the amount of Al, while the product of strength and ductility (RmA-value) increased with increasing the amount of Al. Here it is important to note that Al-1 showed a similar shape memory effect to Al-0 with much improved mechanical properties (ductility, RmA-values).

3.2 Deformation behavior and microstructure

Figure 3 shows SS-curves for the samples Al-0, Al-1, Al-3, and their photos at 3% strain. The SS-curves for the samples Al-2 and Al-3 were similar as well as their optical micrographs.
In the SS-curve of Al-0 (SMA), the boundary between elastic and plastic regions is not clear, and the proof stress is quite low. It should also be noted that work hardening rate is high, for which the plastic region is round (Fig. 3(a)). This is a common feature of the SS-curve where the stress induced $\gamma$-martensitic transformation takes place. The low proof stress is due to the fact that stress-induced $\varepsilon$-martensite occurs at a low stress, and high work hardening is due to an increase in threshold stress to induce transformation as a result of blocking of slip deformation at $\gamma/\varepsilon$ boundaries.\(^{19}\)

On the other hand, high ductility of Fe-Mn-Si-Al-based TWIP steels is attributed to deformation twin.\(^{17,18}\) In the SS-curve of Al-3 (Fig. 3(c)), the yield point is obvious, work hardening rate is low, and the plastic region is flat compared to the SS-curve of Al-0 (Fig. 3(a)). The SS-curve of Al-1 (Fig. 3(b)) has intermediate characteristics of TWIP steel and SMA.

Optical micrographs Fig. 3(d)–(f) corresponding to these three curves also show similar tendency. The deformation microstructures of Al-0 (SMA) have parallel banded relieves along a particular direction (see Fig. 3(d)), which originates from stress-induced $\varepsilon$-martensite.\(^{3,20}\) In this figure, grain boundaries and twin boundaries are indicated by GB and TB. The directions of the martensite platelets are the same in parent phase or twins. This is attributed to the martensite formed on a particular habit plane. In addition, since surface tilt of relieves is almost uniform, they are mainly composed of single martensitic variant formed along the same orientation. On the other hand, deformation microstructure of Al-3 (Fig. 3(f)) also have parallel linear internal microstructures in the region segmented more finely than Fig. 3(d) by grain boundary or annealing twins. But the width is much narrower than Al-0. According to Grassel et al.\(^{20}\) and Vercammen et al.\(^{21}\) parallel linear microstructures in the grains and deformation twins are lamellar deformation twins or dislocation lines. Deformation microstructures of Al-1 as intermediate composition have parallel banded surface relieves in a similar size to Al-0 (Fig. 3(b)) while the sizes of annealing twins lie in between Al-0 and Al-3.

As a consequence, it was found that Al-1 shows intermediate deformation behavior and microstructure between SMA and TWIP steel. Hence, it is probable that both stress-induced $\varepsilon$ martensite and deformation twins are present in the microstructures of Al-1 that shows good SME and high ductility.

3.3 Structural variation by deformation and heating in Fe-30Mn-5Si-1Al alloy

Since the sample Al-1 exhibits SME, it should have stress-induced $\varepsilon$-martensite. We therefore performed TEM and AFM observations of the microstructure of the sample Al-1 during deformation and after heating to confirm this idea.

Figure 4 shows the results of TEM observation of the sample Al-1 deformed by 2% tensile strain. In Fig. 4(a) as bright field image, parallel banded structure is observed. The diffraction patterns of the part surrounded by circle in Fig. 4(a) are shown in Fig. 4(b). Figure 4(c) is the key diagram obtained by analyzing Fig. 4(b), in which the relation of $\gamma$ and $\varepsilon$ are shown.\(^{21}\) One can see that the reflections from $\varepsilon$-martensite appear on the parts shown by arrow in Fig. 4(b). Along the orientation of (111)$_\gamma$ // (0002)$_\varepsilon$, streak lines were observed, showing that stacking faults exist in high concentration. The direction of streak is perpendicular to parallel banded structure in Fig. 4(a), which indicates lamellar $\varepsilon$. These observations confirmed the presence of $\varepsilon$-martensite in the microstructure of deformed Al-1. Thus, the
shape memory effect of the sample Al-1 is attributable to the reverse transformation of stress-induced $\varepsilon$ martensite.

Figs. 5(a) to (c) show AFM micrographs for the sample Al-1 at the tensile strain of 3%, and 10% and that after heating. Surface relieves are observed on the deformed sample surfaces. The image is segmented into three regions due to the formation of annealing twins ($\gamma_T$) in the parent phase ($\gamma_P$). In Fig. 5, TB represents annealing twin boundaries. In $\gamma_P$ and $\gamma_T$, parallel banded surface relieves may be caused by $\varepsilon$-martensite, deformation twins, stacking faults and dislocation lines. However, TEM observations imply that some of them are due to $\varepsilon$-martensite.
After 10% tensile deformation, in the part surrounded by circle the width of parallel banded relieves increased as shown in Fig. 5(b). This part is narrowed after heating as shown in Fig. 5(c).

Figs. 6(a) and (b) show atomic force micrographs of the sample at 10% strain and after heating, respectively. Figure 6(c) shows cross-sectional profiles of surface relieves that are obtained from Figs. 6(a) and (b), in which the width of parallel banded parts is narrowed.

The TEM and AFM observations support the fact that the SME of Al-1 originates from the formation of stress-induced ε martensite on deformation and successive reversed transformation to γ on heating like conventional Fe-Mn-Si alloys. However it is also true that some parallel banded surface relieves remain after heating. They may be caused by deformation twins, sediments of stacking faults or dislocation lines. It is also probable that the remained relieves are caused by γ which transformed from ε on different crystallographic path.\textsuperscript{20)}

4. Summary

We studied the effect of Al content on the SME and the ductility of Fe-Mn-Si alloys. The alloys with Al content of 0% and 1% exhibited similar SME, but no appreciable SME was observed for the alloys with >2%. The ductility increased and the tensile strength decreased with increasing the amount of Al, while their product increased, showing that overall mechanical properties were improved with Al addition. It was interesting to note that Fe-30Mn-5Si-1Al alloy shows relatively good SME and greater ductility (more than 50%) than conventional Fe-Mn-Si shape memory alloys. The SME of Fe-30Mn-5Si-1Al alloy was attributable to reversed transformation of stress-induced ε martensite like conventional Fe-Mn-Si alloys.

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